JC10 Rec' 77/PTO 1 3 FEB 2002

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(REV. 12-2001)	MERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER	
TRANSMITTAL LETTER	TRANSMITTAL LETTER TO THE UNITED STATES		
DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371		U.S. APPLICATION NO. (If known, sec 37 CFR 1.5	
INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED	
PCT/EP00/07524	03 August 2000 (3.08.00)	16 August 1999 (16.08.99)	
TITLE OF INVENTION ANTISTATIC AGENT			
APPLICANT(S) FOR DO/EO/US DOBLI	ER, Martin; KOHLER, Walter; BIER	, Peter; EBERT, Wolfgang;	
GORNY, Rudiger and NEUMANN, Applicant herewith submits to the United St	stegmed tates Designated/Elected Office (DO/EO/US)	the following items and other information:	
1. X This is a FIRST submission of item			
	NT submission of items concerning a filing		
3. X This is an express request to begin ritems (5), (6), (9) and (21) indicated	national examination procedures (35 U.S.C. 3	371(f)). The submission must include	
4. X The US has been elected by the exp	iration of 19 months from the priority date (A	Article 31).	
5. X A copy of the International Applica	tion as filed (35 U.S.C. 3/1(c)(2)) and only if not communicated by the Internation	onal Bureau).	
	by the International Bureau.	,	
c. is not required, as the app	lication was filed in the United States Receive	ving Office (RO/US).	
6. An English language translation of	the International Application as filed (35 U.S	S.C. 371(c)(2)).	
a. X is attached hereto.			
b. has been previously subm	nitted under 35 U.S.C. 154(d)(4).	(25 H C C 271(c)(3))	
7. Amendments to the claims of the In	nternational Aplication under PCT Article 19 red only if not communicated by the Internat	tional Bureau)	
	by the International Bureau.	Date Date Care y	
	ever, the time limit for making such amendn	cents has NOT expired	
1.1 · · · · · · · · · · · · · · · · · ·		iens has ito i expired.	
d. have not been made and v		#-1- 10 (25 H.C.C. 271 (a)(2))	
13	the amendments to the claims under PCT Ar	TICLE 19 (35 U.S.C. 3/1 (C)(3)).	
9. X An oath or declaration of the invent			
10. An English lanugage translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).			
Items 11 to 20 below concern docume			
	ment under 37 CFR 1.97 and 1.98.		
12. X An assignment document for reco	ording. A separate cover sheet in compliance	e with 37 CFR 3.28 and 3.31 is included.	
13. X A FIRST preliminary amendmen	at.	,	
14. A SECOND or SUBSEQUENT	preliminary amendment.		
15. A substitute specification.			
16. A change of power of attorney at			
	e sequence listing in accordance with PCT Re		
	international application under 35 U.S.C. 154		
19. A second copy of the English lan	nguage translation of the international applic	ation under 35 U.S.C. 154(d)(4).	
20. X Other items or information:			
PTO Form 1449 w/references listed	thereon		

11 Rec'd PCT/PTO 1 3 FEB 2002 STERNATIONAL APPLICATION NO Mo-6954/LeA 33,936 C/EP00/07524 CALCULATIONS PTO USE ONLY 21.X The following fees are submitted: BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) -(5)): Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO. \$1040.00 International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO . . . International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(I)-(4) . . . . . . . \$710.00 International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) . . ENTER APPROPRIATE BASIC FEE AMOUNT 890.00 Surcharge of \$130.00 for furnishing the oath or declaration later than S months from the earliest claimed priority date (37 CFR 1.492(e)). 9 RATE NUMBER FILED NUMBER EXTRA CLAIMS x \$18.00 \$ 0.00 - 20 = 0 Total claims \$ 0.00 x \$84.00 Independent claims -3 = \$ + \$280.00 MULTIPLE DEPENDENT CLAIM(S) (if applicable) \$ TOTAL OF ABOVE CALCULATIONS Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above S are reduced by 1/2. \$ 890.00 Processing fee of \$130.00 for furnishing the English translation later than \$ months from the earliest claimed priority date (37 CFR 1.492(f)). TOTAL NATIONAL FEE 8 Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property \$ 40.00 \$ 930.00 TOTAL FEES ENCLOSED Amount to be refunded: S \$ charged: \_\_\_\_ to cover the above fees is enclosed. a. A check in the amount of \$ \_\_ Please charge my Deposit Account No.  $\underline{13-384}8$ \_\_\_ in the amount of \$ 930.00 to cover the above fees. A duplicate copy of this sheet is enclosed. c. X The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 13-3848 A duplicate copy of this sheet is enclosed. Fees are to be charged to a credit card. WARNING: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038. NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status. SEND ALL CORRESPONDENCE TO: Aron Preis SIGNATURE Aron Preis

NAME

29,426 REGISTRATION NUMBER

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1U/U4782/ JC11 Rec'd PGT/PTO 13 FEB 2002

PATENT APPLICATION Mo-6954 LeA 33.936

# IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICATION OF		) ) PCT/EP00/07524 )
MARTIN DÖBLER ET AL		
SERIAL NUMBER: TO BE ASSIGNED		{
FILED:	HEREWITH	{
TITLE:	ANTISTATIC AGENT	) }

# PRELIMINARY AMENDMENT

Assistant Commissioner for Patents Washington, D.C. 20231

Sir:

THE TRANSPORT OF THE OWNER, WHITE CO.

Prior to its examination kindly amend the enclosed translation of the captioned patent application as follows:

"Express Mail" mailing label number <u>ET700176412US</u>
Date of Deposit February 13, 2002
I hereby certify that this paper or fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to the Assistant Commissioner of Patents and Trademarks, Washington, D.C. 20231

(Name of person maling paper or fee)

Signature of person maling paper or fee)

### IN THE SPECIFICATION:

Add page 15, a page containing an abstract that reads as follows:

### -- ANTISTATIC AGENT

### ABSTRACT OF THE DISCLOSURE

An agent suitable for imparting antistatic properties to plastics is disclosed. The incorporation of small amounts of perfluoroalkylsulfonic acid salt in plastic resins, notably thermoplastic resins was found to be effective for this purpose.—

## IN THE CLAIMS:

Please amend as follows:

Cancel Claims 1-7.

Add the following:

- --8. A thermoplastic molding composition comprising a thermoplastic resin and an amount of perfluoroalkylsulfonic acid salt sufficient to render the composition antistatic.
- The molding composition of Claim 8 characterized in that it is transparent.
  - 10. The molding composition of Claim 8 wherein the salt conforms to

#### RA-SO<sub>3</sub> X

wherein R denotes perfluorinated linear or branched carbon chain having 1 to 30 carbon atoms, A denotes a direct bond or an aromatic nucleus, and X denotes an alkylated and/or arylated ammonium ion NR'R"R"", phosphonium ion

RR'R"R"", sulfonium ion SR'R"R""R", imidazolinium ion, pyridinium ion, or tropylium ion

wherein R', R", R" and R"" independently one of the others denote halogenated or non halogenated, linear or branched carbon chains having 1 to 30 carbon atoms.

11. The molding composition of Claim 8 wherein the perfluoroalkylsulfonic acid salt is at least one member selected from the group consisting of perfluorooctanesulfonic acid tetraethylammonium salt. perfluorobutanesulfonic acid tetraethylammonium salt. perfluorooctanesulfonic acid tetrabutylphosphonium salt. perfluorobutanesulfonic acid tetrabutylphosphonium salt. perfluorooctanesulfonic acid benzyltrimethylammonium salt. perfluorobutanesulfonic acid benzyltrimethylammonium salt, perfluorooctanesulfonic acid trimethylphenylammonium salt, perfluorobutanesulfonic acid trimethylphenylammonium salt. perfluorobutanesulfonic acid dimethyldiphenylammonium salt, perfluorooctanesulfonic acid dimethyldiphenylammonium salt, perfluorobutanesulfonic acid trimethylneopentylammonium salt, perfluorooctanesulfonic acid trimethylneopentylammonium salt. perfluorobutanesulfonic acid dimethyldineopentylammonium salt. perfluorooctanesulfonic acid dimethyldineopentylammonium salt, perfluorobutanesulfonic acid tetrabutylphosphonium salt, and perfluorooctanesulfonic acid tetrabutylphosphonium salt.

- 12. The molding composition of Claim 8 wherein the amount is 0.01 to 2 percent relative to the weight of the composition.
- 13. The molding composition of Claim 8 wherein the resin is at least one member selected from the group consisting of (co)polycarbonate, (co)polyacrylate, (co)polymethacrylate, (co)polystyrene, transparent polyurethane, polyolefin and (co)polycondensation products of terephthalic acid.

- The molding composition of Claim 8 wherein the resin is (co)polycarbonate.
- The molding composition of Claim 8 wherein the salt is perfluorooctanesulfonic acid tetraethylammonium.
- The molding composition of Claim 15 wherein the resin is (co)polycarbonate.
- 17. A method of imparting antistatic properties to a thermoplastic resin comprising incorporating therewith perfluoroalkylsulfonic acid salt in an amount of 0.01 to 2 percent relative to the weight of the composition.
  - 18. A molded article comprising the molding composition of Claim 8.--

### REMARKS

The present amendment seeks to place the application in better conformance with U.S. practice. A page containing an Abstract of the Disclosure is enclosed. Entry of the amendment is requested.

Respectfully submitted,

By\_

Aron Preis Attorney for Applicants Reg. No. 29,426

Bayer Corporation 100 Bayer Road Pittsburgh, Pennsylvania 15205-9741 (412) 777-8343 FACSIMILE PHONE NUMBER: (412) 777-8363

s:/sr/ap0256

#### VERSION WITH MARKINGS TO SHOW CHANGES MADE:

### IN THE SPECIFICATION:

Add page 15, a page containing an abstract that reads as follows:

### -- ANTISTATIC AGENT

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An agent suitable for imparting antistatic properties to plastics is disclosed. The incorporation of small amounts of perfluoroalkylsulfonic acid salt in plastic resins, notably thermoplastic resins was found to be effective for this purpose.—

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Cancel Claims 1-7.

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  - 10. The molding composition of Claim 8 wherein the salt conforms to

RA-SO<sub>3</sub> X

wherein R denotes perfluorinated linear or branched carbon chain having 1 to 30 carbon atoms. A denotes a direct bond or an aromatic nucleus, and X denotes an

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alkylated and/or arylated ammonium ion NR'R"R""R"", phosphonium ion RR'R"R"R"", sulfonium ion SR'R"R""R"", imidazolinium ion, pyridinium ion, or tropylium ion

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- 12. The molding composition of Claim 8 wherein the amount is 0.01 to 2 percent relative to the weight of the composition.
- 13. The molding composition of Claim 8 wherein the resin is at least one member selected from the group consisting of (co)polycarbonate, (co)polyacrylate, (co)polymethacrylate, (co)polystyrene, transparent polyurethane, polyolefin and (co)polycondensation products of terephthalic acid.

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- The molding composition of Claim 8 wherein the resin is (co)polycarbonate.
- The molding composition of Claim 8 wherein the salt is perfluorooctanesulfonic acid tetraethylammonium.
- The molding composition of Claim 15 wherein the resin is (co)polycarbonate.
- 17. A method of imparting antistatic properties to a thermoplastic resin comprising incorporating therewith perfluoroalkylsulfonic acid salt in an amount of 0.01 to 2 percent relative to the weight of the composition.
  - 18. A molded article comprising the molding composition of Claim 8.-

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Date of Deposit February 13, 200

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1.10 on the date francische developments, Washington, D. 2001/04985

Donna J. Ventrol 0/04985

(Name of person reality paper or fee)

#### Antistatic agent

JC11 Res'd FCT/PTO 1 3 FEB 2002

This application relates to the use of fluorinated alkylsulfonic acids salts as an antistatic agent in particular in plastics and to plastics containing fluorinated alkylsulfonic acid salts and to mouldings producible therefrom.

The deposition of dust to form dust figures is a widespread problem with plastics mouldings, c.f. in this connection for example Saechtling Kunststoff-Taschenbuch, 26th edition, Hanser Verlag, 1995, Munich, pp. 140 et seq.. Dust deposits on transparent mouldings are particularly troublesome and restrict function. Such mouldings are used, for example, for applications in optical data storage media, electrical engineering, automotive engineering, in the building sector, for liquids containers or for other optical applications. Dust deposition is undesirable for all these applications and may impair function.

One known method for reducing the deposition of dust on plastics articles is to use antistatic agents. The literature contains descriptions of antistatic agents for thermoplastics which restrict dust deposition (c.f. for example Gächter, Müller, Plastic Additives, Hanser Verlag, Munich, 1996, pp. 749 et seq.). These antistatic agents improve the electrical conductivity of the plastics moulding compositions and thus dissipate any surface charges which develop during production and use. Dust particles are thus less attracted and dust deposition is consequently reduced.

A distinction is generally drawn between internal and external antistatic agents. An external antistatic agent is applied onto the plastics moulding after processing, while an internal antistatic agent is added as an additive to the plastics moulding compositions. On economic grounds, it is usually desirable to use internal antistatic agents, as no further operations for applying the antistatic agent are required after processing. Few internal antistatic agents have hitherto been described in the literature which also form completely transparent mouldings, especially with polycarbonate. JP-06228420 A 940816 describes aliphatic sulfonic acid ammonium salts as an antistatic agent in polycarbonate. However, these compounds bring about

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a reduction in molecular weight. JP-62230835 describes the addition of 4% of nonylphenylsulfonic acid tetrabutylphosphonium to polycarbonate.

One disadvantage of known antistatic agents is that they must be used in relatively high concentrations in order to achieve the antistatic effect. However, such addition modifies the material properties of the plastics in an unwanted manner.

The object of the invention is accordingly to provide antistatic agents which have no negative impact on the material properties of plastics.

It has surprisingly been found that perfluoroalkylsulfonic acid salts are particularly suitable as antistatic agents for the production of injection moulded and extruded mouldings. Even small quantities of perfluoroalkylsulfonic acid salt give rise to mouldings which no longer attract dust.

This application accordingly provides the use of perfluoroalkylsulfonic acid salts as antistatic agents, in particular for plastics, in particular for transparent plastics, as well as plastics, plastics moulding compositions and plastics mouldings containing at least one perfluoroalkylsulfonic acid salt.

Preferably suitable perfluoroalkylsulfonic acid salts are salts of the type (I)

RA-SO<sub>3</sub> X (I)

25 in which

- R means perfluorinated linear or branched carbon chains having 1 to 30 carbon atoms, preferably 4 to 8 carbon atoms;
- 30 A means a direct bond or an aromatic nucleus, for example and preferably fluorinated or non-fluorinated o-, m- or p-phenylene;

X means an alkylated and/or arylated ammonium ion NRR"R""R"", phosphonium ion PR'R"R"", sulfonium ion SR'R"R", and a substituted or unsubstituted imidazolinium ion, pyridinium ion or tropylium ion, in which R', R", R"", mutually independently denote halogenated or non-halogenated, linear or branched carbon chains having 1 to 30 carbon atoms, preferably 1 to 4 carbon atoms, in particular methyl, ethyl, propyl, butyl, pentyl, hexyl, isopropyl, isobutyl, tert.-butyl, neopentyl or aromatic residues or alkylaromatic residues such as for example and preferably phenyl, benzyl, alkylphenyl in each case having 1 to 4 carbon atoms in the alkyl portion.

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#### The following are preferred:

- perfluorooctanesulfonic acid tetraethylammonium salt,
- perfluorobutanesulfonic acid tetraethylammonium salt,
- perfluorooctanesulfonic acid tetrabutylphosphonium salt,
- perfluorobutanesulfonic acid tetrabutylphosphonium salt,
  - perfluorooctanesulfonic acid benzyltrimethylammonium salt,
  - perfluorobutanesulfonic acid benzyltrimethylammonium salt,
  - perfluorooctanesulfonic acid trimethylphenylammonium salt,
  - perfluorobutanesulfonic acid trimethylphenylammonium salt,
  - perfluorobutanesulfonic acid dimethyldiphenylammonium salt,
  - perfluorooctanesulfonic acid dimethyldiphenylammonium salt,
  - perfluorobutanesulfonic acid trimethylneopentylammonium salt,
  - perfluorooctanesulfonic acid trimethylneopentylammonium salt,
  - perfluorobutanesulfonic acid dimethyldineopentylammonium salt,

perfluorooctanesulfonic acid dimethyldineopentylammonium salt.

- perfluorobutanesulfonic acid tetrabutylphosphonium salt,
- perfluorooctanesulfonic acid tetrabutylphosphonium salt.

Mixtures of sulfonic acid salts, in particular of the above-stated sulfonic acid salts, are also preferred.

Perfluorooctanesulfonic acid tetraethylammonium salt is particularly preferred.

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Perfluoroalkylsulfonic acid salts are known or may be produced using known methods. The salts of the sulfonic acids may be produced by combining equimolar quantities of the free sulfonic acid with the hydroxy form of the corresponding cation in water at room temperature and evaporating the solution.

- 4 -

The perfluoroalkylsulfonic acids are preferably added to the plastics in quantities of 0.001 to 2 wt.%, preferably of 0.1 to 1 wt.%.

Plastics are preferably taken to mean thermoplastics, in particular transparent thermoplastics, preferably the polymers of ethylenically unsaturated monomers and/or polycondensation products of diffunctional reactive compounds.

Particularly suitable plastics are polycarbonates or copolycarbonates based on diphenols, poly- or copolyacrylates and poly- or copolymethacrylates such as for example and preferably polymethyl methacrylate, poly- or copolymers with styrene such as for example and preferably transparent polystyrene or styrene/acrylonitrile copolymer (SAN), transparent thermoplastic polyurethanes, and polyolefins, such as for example and preferably transparent grades of polypropylene or polyolefins based on cyclic olefins (for example TOPAS®, Hoechst), poly- or copolycondensation products of terephthalic acid, such as for example and preferably poly- or copolyethylene terephthalate (PET or CoPET) or glycol-modified PET (PETG).

Polycarbonates or copolycarbonates are particularly preferred, in particular non-halogenated polycarbonates and/or copolycarbonates having molecular weights  $\overline{M}_{\rm w}$  of 500 to 100,000, preferably of 10,000 to 50,000, particularly preferably of 15,000 to 40,000.

For the purposes of the present invention, thermoplastic, aromatic polycarbonates comprise both homopolycarbonates and copolycarbonates; the polycarbonates may be linear or branched in known manner.

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2,2-bis-(3,5-dichloro-4-

The polycarbonates according to the invention may also be present in entirely or partially brominated form.

These polycarbonates are produced in known manner from diphenols, carbonic acid derivatives, optionally chain terminators and optionally branching agents.

Details of the production of polycarbonates have been described in many patents over the last 40 years or so. Reference is made purely by way of example to Schnell, "Chemistry & Physics of Polycarbonates", Polymer Reviews, vol. 9, Interscience Publishers, New York, London, Sydney 1964, to D. Freitag, U. Grigo, P.R. Müller, H. Nouvertne', Bayer AG, "Polycarbonates" in Encyclopedia of Polymer Science & Engineering, vol. 11, 2nd edition, 1988, pp. 648-718 and finally to Dr. U. Grigo, Dr. K. Kirchner and Dr. P.R. Müller, "Polycarbonate" in Becket/Braun, Kunststoff-Handbuch, vol. 3/1, Polycarbonate, Polyacetale, Polyester, Celluloseester, Carl Hanser Verlag, Munich/Vienna, 1992, pp. 117-299.

 $\label{eq:continuous} 4.4'-dihydroxyphenyl), 2.2-bis-(4-hydroxyphenyl)-p-diisopropylbenzene, 2.2-bis-(3-methyl-4-hydroxyphenyl)-p-diisopropylbenzene, 2.2-bis-(3-methyl-4-hydroxyphenyl)propane, 2.2-bis-(3-chloro-4-hydroxyphenyl)propane, bis-(3,5-dimethyl-4-hydroxyphenyl)methane, 2.2-bis-(3,5-dimethyl-4-hydroxyphenyl) sulfone, 2.4-bis-(3,5-dimethyl-4-hydroxyphenyl)-2-methylbutane, 1,1-bis-(3,5-dimethyl-4-hydroxyphenyl)-2-methylbutane, 1,1-bis-(3,5$ 

Preferred diphenols for the production of polycarbonates are:

hydroxyphenyl)-p-diisopropylbenzene,

25 hydroxyphenyl)propane, 2,2-bis-(3,5-dibromo-4-hydroxyphenyl)propane and 1,1-bis-(4-hydroxyphenyl)-3,3,5-trimethylcyclohexane.

Particularly preferred diphenols are 2,2-bis-(4-hydroxyphenyl)propane, 2,2-bis-(3,5-dimethyl-4-hydroxyphenyl)propane, 2,2-bis-(3,5-dichloro-4-hydroxyphenyl)propane, 2,2-bis-(3,5-dibloromo-4-hydroxyphenyl)propane, 1,1-bis-(4-hydroxyphenyl)-3,3,5-trimethylcyclohexane and 1,1-bis-(4-hydroxyphenyl)-3,3,5-trimethylcyclohexane.

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Preferred branching agents are triphenols, trimesic acid (trichloride), cyanuric acid trichloride and 3.3-bis-(3-methyl-4-hydroxyphenyl)-2-oxo-2.3-dihydroindole.

Improved plastics compositions may be obtained by incorporating at least one further additive conventionally present in thermoplastics, preferably poly- and copolycarbonates, such as for example stabilisers (as described, for example, in EP 0 839 623 A1 or EP 0 500 496 A1), especially heat stabilisers, in particular organic phosphites or phosphines, for example and preferably triphenylphosphine, mould release agents, for example and preferably fatty acid esters of glycerol or tetramethanolmethane, wherein unsaturated fatty acids may be entirely or partially epoxidised, in particular glycerol monostearate or pentaerythritol tetrastearate (PETS), flame retardants, UV absorbents, for example and preferably hydroxybenzotriazoles and hydroxytriazines, fillers, foaming agents, dyes, pigments, optical brighteners, transesterification catalysts and nucleating agents or the like, preferably in quantities of in each case up to 5 wt.%, preferably of 0.01 to 5 wt.%, relative to the entire mixture, particularly preferably of 0.01 wt.% to 1 wt.%, relative to the quantity of plastics.

The perfluoroalkylsulfonic acid salts and optionally the additives or mixtures of additives are generally incorporated in the conventional manner for example before or during polymerisation or by subsequent mixing with the plastic.

The plastics compositions obtained in this manner are generally in the form of solutions, dispersions, emulsions, finely divided solids, powders, pellets, platelets or flakes (moulding compositions) and are used for the production of shaped articles (mouldings).

Shaped articles are for example and preferably light-transmitting articles such as for example and preferably light diffusers for motor vehicles, lenses, such as for example spectacle lenses, films, tapes, sheets, multi-wall sheets, multiple wall sheets, containers, tubes and other profiles which are produced using conventional

methods, such as for example hot pressing, spinning, extrusion or injection moulding. The polymer compositions may also be converted into cast films.

It is also of interest to use the plastics composition according to the invention for the production of multilayer systems. In such cases, the plastics composition according to the invention is applied in a thin layer onto a shaped article of a plastic which does not have an antistatic finish or additive. Application may proceed simultaneously with or immediately after shaping of the moulding, for example by coextrusion or multi-component injection moulding. Application may, however, also be performed onto the pre-shaped substrate, for example by lamination with a film or by coating with a solution.

It is furthermore of interest to use the plastics compositions according to the invention for the production of casings for electrical and electronic appliances, such as for example televisions, monitors, computers, printers, mobile telephones, clocks, hi-fi systems and the like in any desired colours with a transparent, translucent or opaque formulation, optionally with a flame-retardant finish or additive.

Plastics compositions containing perfluoroalkylsulfonic acid ammonium salts are preferably used for the production of light diffusers for motor vehicles.

It is also of particular interest to use plastics compositions containing perfluoroalkylsulfonic acid salts for the production of sheet, double-wall sheet, coextruded sheet and film.

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The advantage of the plastics mouldings provided with the antistatic agents according to the invention is that these mouldings no longer develop a static charge, for example during production, when the conventionally used protective films are peeled off or during transport and storage.

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The following Examples illustrate the invention. The invention is not limited to the Examples. The percentages stated below are weight percentages.

- 8 -

#### Dust test

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In order to test dust deposition in a laboratory test, the injection moulded sheets are exposed to an atmosphere containing suspended dust. To this end, a 2 litre beaker containing an 80 mm long magnetic stirrer rod of a triangular cross-section is filled to a depth of approx. 1 cm with dust (coal dust/20 g activated carbon, Riedel de Haen, Seelze, Germany, item no. 18003). The dust is suspended in the atmosphere with a magnetic stirrer. Once the stirrer has been shut off, the test specimen is exposed to this dusty atmosphere for 7 seconds. Depending upon the test specimen used, a greater or lesser amount of dust is deposited on the test specimens.

Evaluation of dust deposition (dust figures) is performed visually. Sheets exhibiting dust figures were rated (-), while virtually dust-free sheets were rated (+).

# 15 Example 1

In order to produce the test specimens, an additive-free, unstabilised polycarbonate (Makrolon® 2808 from Bayer AG, Leverkusen) having an average molecular weight of approx. 30,000 ( $M_w$  by GPC), solution viscosity:  $\eta=1.293$  at 340°C, is compounded in a twin screw extruder with the quantity of perfluoroctanesulfonic acid tetraethylammonium salt (Bayowet 248® from Bayer AG, Leverkusen) and the other stated additives shown in Table 1 and then pelletised.

Rectangular sheets (155 mm x 75 mm x 2 mm) are then injection moulded from these pellets at various melt temperatures and subjected to the dust test. The results are stated in Table 2.

Table 1: Plastics compositions

Example	Composition		
1.1	1% Bayowet 248® + 0.025% triphenylphosphine + 0.3% 2-(2'-hydroxy-		
	3'-(2-butyl)-5'-(tert-butyl)phenyl)benzotriazole (Tinuvin® 350, Ciba		
	Spezialitätenchemie, Basel)		
1.2	0.6% Bayowet 248® + 0.025% triphenylphosphine + 0.3% 2-(2'-		
	hydroxy-3'-(2-butyl)-5'-(tert-butyl)phenyl)benzotriazole		
1.3	0.4% Bayowet 248® + 0.025% triphenylphosphine + 0.3% 2-(2'-		
	hydroxy-3'-(2-butyl)-5'-(tert-butyl)phenyl)benzotriazole		
1.4	0.3% Bayowet 248® + 0.025% triphenylphosphine + 0.3% 2-(2'-		
	hydroxy-3'-(2-butyl)-5'-(tert-butyl)phenyl)benzotriazole		
1.5	0.25% Bayowet 248® + 0.025% triphenylphosphine + 0.3% 2-(2'-		
	hydroxy-3'-(2-butyl)-5'-(tert-butyl)phenyl)benzotriazole		
1.6	0.2% Bayowet 248® + 0.025% triphenylphosphine + 0.3% 2-(2'-		
	hydroxy-3'-(2-butyl)-5'-(tert-butyl)phenyl)benzotriazole		
1.7	0.15% Bayowet 248® + 0.025% triphenylphosphine + 0.3% 2-(2'-		
	hydroxy-3'-(2-butyl)-5'-(tert-butyl)phenyl)benzotriazole		
1.8	0.1% Bayowet 248® + 0.025% triphenylphosphine + 0.3% 2-(2'-		
	hydroxy-3'-(2-butyl)-5'-(tert-butyl)phenyl)benzotriazole		

All the colour sample sheets produced from the plastics compositions of Examples 1.1 to 1.8 at melt temperatures a), b) and c) are completely transparent on visual inspection.

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Table 2: Results of dust test

Example	a) 300°C	b) 320°C	e) 330°C
1.1	+		
1.2	+		
1.3	+	+	+
1.4	-	+	+
1.5	-	+	+
1.6	-	+	+
1.7	-	+	+
1.8	-	-	+

# Example 2:

Polycarbonate films of a thickness of 0.25 mm based on the polycarbonate Makrolon 3100® from Bayer AG, Leverkusen and the antistatic agent contents stated in Table 3 are produced by extrusion at a melt temperature of 280°C. Antistatic action is determined by measuring surface conductivity to DIN IEC 93  $(\Omega)$ .

Table 3: Plastics film composition

Example	Composition	Dust test	Surface conductivity
2.1	No additions	-	$1.010^{17}\Omega$
2.2	0.3% Bayowet 248®	+	2.7 10 <sup>15</sup> Ω
2.3	0.5% Bayowet 248®	+	8.2 10 <sup>13</sup> Ω
2.4	1% Bayowet 248®	+	2.9 10 <sup>13</sup> Ω
2.5	1.5% Bayowet 248®	+	$6.0\ 10^{12}\Omega$

Surface resistance is reduced by almost 2 orders of magnitude by the addition of 0.3 wt.% of Bayowet® 248 and by more than 4 orders of magnitude by the addition of 1.5 wt.%. This is significantly better than the values hitherto described for antistatic agents in this range of concentrations in polycarbonate.

## Example 3:

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The compositions stated in Table 4 are produced according to Example 1 and subjected to the dust test. The perfluorobutanesulfonic acid and perfluorocatanesulfonic acid may be liberated by the action of concentrated sulfuric acid on the potassium salts of the sulfonic acids and isolated by distillation. The potassium salts are obtainable from Aldrich or from Bayer AG, Leverkusen. Trimethylphenylammonium hydroxide may be produced from trimethylphenylammonium chloride (Aldrich) by means of ion exchange on the anion exchanger Lewatit® 500 (Bayer AG). Tetraethylammonium hydroxide and benzyltrimethylammonium hydroxide are obtainable from Aldrich.

Table 4: Plastics compositions

Example	Composition	Melt	Dust test
		temperature	
3.1	0.3% perfluorobutanesulfonic acid	320°C	+
	tetraethylammonium salt + 0.025%		
	triphenylphosphine + 0.3% 2-(2'-hydroxy-3'-		
	(2-butyl)-5'-(tert-butyl)phenyl)benzotriazole		
3.2	0.3% perfluorobutanesulfonic acid	320°C	+
	benzyltrimethylammonium salt + 0.025%		
	triphenylphosphine + 0.3% 2-(2'-hydroxy-3'-		
	(2-butyl)-5'-(tert-butyl)phenyl)benzotriazole		
3.3	0.3% perfluorooctanesulfonic acid	300°C	+
	trimethylphenylammonium salt + 0.025%		
	triphenylphosphine + 0.3% 2-(2'-hydroxy-3'-		
	(2-butyl)-5'-(tert-butyl)phenyl)benzotriazole		
3.4	1% perfluorooctanesulfonic acid	300°C	+
	tetraethylammonium salt + 1% titanium		
	dioxide Cronos C12230 + 0.2%		
	perfluorobutanesulfonic acid potassium salt		
	(Bayer) + 0.09% Teflon 6CN (DuPont)		
3.5	1% perfluorooctanesulfonic acid	300°C	+
	tetraethylammonium salt + 1% titanium		
	dioxide Cronos C12230		

# Example 4:

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In a similar manner to Example 1, plastics compositions are produced from Bayblend® (blend of ABS and bisphenol A polycarbonate of Bayer AG), Apec® (copolycarbonate of Bayer AG) and Pocan® (unreinforced polybutylene terephthalate of Bayer AG) with the quantities of antistatic agent and at the melt

temperatures stated in Table 5 and were subjected to the dust test. The results are given in Table 5.

Table 5: Action of sulfonic acid salts in further thermoplastics

Example	Composition	Melt	Dust
		temperature	test
4.1	Bayblend FR2000® + 3% Bayowet 248®	250°C	+
4.2	Bayblend FR2000® + 1% Bayowet 248®	250°C	+/-
4.3	Bayblend T45® + 3% Bayowet 248®	270°C	+
4.4	Pocan B1305® + 1% Bayowet 248®	260°C	+
4.5	Apec® HT KU1-9201=9330 + 0.5% Bayowet 248®	300°C	+
4.6	Apec® HT KU1-9201=9330 + 0.5% Bayowet 248®	340°C	+

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### Patent Claims

- 1. Use of perfluoroalkylsulfonic acid salts as an antistatic agent.
- Use of perfluoroalkylsulfonic acid salts to impart antistatic properties to plastics.
  - 3. Plastics compositions containing at least one perfluoroalkylsulfonic acid salt.
- 10 4. Process for the production of plastics compositions as defined in claim 3, characterised in that at least one perfluoroalkylsulfonic acid salt is added before, during or after polymerisation of the plastics.
  - Use of plastics compositions containing perfluoroalkylsulfonic acid salt for the production of mouldings.
  - Process for the production of mouldings having antistatic properties, characterised in that at least one plastics composition as defined in claim 3 is used as the starting material.
  - 7. Plastics mouldings containing at least one perfluoroalkylsulfonic acid salt.

# ANTISTATIC AGENT

# ABSTRACT OF THE DISCLOSURE

An agent suitable for imparting antistatic properties to plastics is disclosed. The incorporation of small amounts of perfluoroalkylsulfonic acid salt in plastic resins, notably thermoplastic resins was found to be effective for this purpose.

#### COMBINED DECLARATION AND POWER OF ATTORNEY

ATTORNEY DOCKET NO

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought

on the invention entitled

#### ANTISTATIC AGENT

the specification of which is attached hereto,

or was filed on August 3, 2000

as a PCT Application Serial No. PCT/EP00/07524

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, \$1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, \$119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s), the priority(ies) of which is/are to be claimed:

199 38 735.4 (Number)

Germany (Country) August 16, 1999

(Month/Day/Year Filed)

199 43 637.1 (Number) Germany (Country)

September 13, 1999 (Month/Day/Year Filed)

I hereby claim the benefit under Title 35, United States Code, \$120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, \$112, I acknowledge the duty to disclose the material information as defined in Title 37, Code of Federal Regulations, \$1.56 which occured between the filling date of the prior application and the national or PCT international filling date of this application:

(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
(Application Serial No.)	(Filing Date)	(Status)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

中門型山山 POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and this application and to transact all business in the Patent and Trademark Office conne JOSEPH C. GIL, Patent Office Registration Number 26.602 ARON PREIS, Patent Office Registration Number 29,426 LYNDANNE M. WHALEN. Patent Office Registration Number 29,457 THOMAS W. ROY. Patent Office Registration Number 29,582 RICHARD E. L. HENDERSON, Patent Office Registration Number 31.619 GODFRIED R. AKORLI, Patent Office Registration Number-28,779 N. DENISE BROWN, Patent Office Registration Number 36,097 NOLAND J. CHEUNG, Patent Office Registration Number 39,138 DIDERICO VAN EYL, Patent Office Registration Number 38,641 CAROLYN M. SLOANE, Patent Office Registration Number 44,339 JAMES R. FRANKS, Patent Office Registration Number 42,552 JACKIE ANN ZURCHER, Patent Office Registration Number 42,251 RAYMOND J. HARMUTH, Patent Office Registration Number 33.896 all of Bayer Corporation, Pittsburgh, Pennsylvania 15205-9741 Send Correspondence To: Direct Telephone Calls To: Patent Department Bayer Corporation (412) 777-2349 100 Baver Road Pittsburgh, Pennsylvania 15205-9741 FULL NAME OF SOLE OR FIRST INVENTOR 2002-01-09 Martin Döbler CITIZENSHIP RESTDENCE D 40593 Düsseldorf, Germany German POST OFFICE ADDRESS c/o Bayer Aktiengesellschaft, D 51368 Leverkusen, Germany INVENTOR'S SIGNATURE FULL NAME OF SECOND INVENTOR 7007-01-02 Walter Köhler CITIZENSHIP RESIDENCE German D 47239 Duisburg, Germany POST OFFICE ADDRESS c/o Bayer Aktiengesellschaft, D 51368 Leverkusen, Germany DATE INVENTOR'S SIGNATURE FULL NAME OF THIRD INVENTOR 2002-01-09 Peter Bier CITIZENSHIP RESIDENCE German D 47800 Krefeld, Germany JAX POST OFFICE ADDRESS c/o Bayer Aktiengesellschaft, D 51368 Leverkusen, Germany DATE FULL NAME OF FOURTH INVENTOR 2002-01-09 Wolfgang Ebert RESIDENCE German D 47800 Krefeld, Germany DFX POST OFFICE ADDRESS c/o Bayer\_Aktiengesellschaft, D 51368 Leverkusen, Germany INVENTOR'S SIGNATURE FULL NAME OF FIFTH INVENTOR 2002-01-0 Rüdiger Gorny CITIZENSHI RESIDENCE German D 47800 Krefeld, Germany DEX POST OFFICE ADDRESS c/o Bayer Aktiengesellschaft, D 51368 Leverkusen, Germany INVENTOR'S SIGNATURE FULL NAME OF SIXTH INVENTOR Jullins 2002-01-10 Siegfried Neumann CITIZENSHIE RESTDENCE D 47918 Tönisvorst, Germany DEX German POST OFFICE ADDRESS c/o Bayer Aktiengesellschaft, D 51368 Leverkusen, Germany DATE INVENTOR'S SIGNATURE FULL NAME OF SEVENTH INVENTOR

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